

# The influence of thermo-mechanical treatment on the microstructure of Fe-30Ni alloy

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## Abstract

The Fe-30Ni alloy investigated exhibits a single-phase austenitic structure. In this alloy the martensitic transformation can occur both during quenching and plastic deformation.

Thermo-mechanical treatments applied for Fe-30Ni can result in the formation of a wide range of microstructures with various martensite morphologies and volume fractions, what gives a possibility to achieve different properties of the alloy. In the present work a microstructural analysis of the deformed Fe-30Ni alloy was performed after several variants of thermo-mechanical treatment. The microstructure was examined using light microscopy (LM) and transmission electron microscopy (TEM). The determination of the volume fraction of martensite formed in different thermo-mechanical treatments was performed using stereological methods and magnetic measurements. The mechanical properties of the alloy investigated were determined in tensile tests.

**Key words:** iron alloys, deformation structure, martensitic phase transformation, light microscopy, transmission electron microscopy, rolling

## 1. Introduction

Deformation induced martensitic transformation in thermodynamically metastable metallic phases was investigated by many research groups, also in the field of fundamental science [1–8]. The Fe-30Ni alloy is of special interest, due to its properties and applications. After the solution treatment the Fe-30Ni alloy exhibits a metastable austenitic structure, which can be transformed into a martensitic one, both by deformation or quenching. The thermo-mechanical treatment applied enables to transform the Fe-30Ni alloy into a composite-like material, with exceptional structure, texture and mechanical properties. In composite-like materials the preferentially oriented strengthening phase exerts a strong influence on mechanical properties. It is the martensite that is a strengthening phase in the Fe-30Ni alloy. Thermo-mechanical treatments applied to the Fe-30Ni alloy can form a wide range of

microstructures with various martensite morphologies and volume fractions.

Rolling at room temperature (RT) leads to the formation of shear bands. After perpendicular rolling, when the secondary path is turned by 90° around primary longitudinal rolling direction, transcrystalline slip bands are formed. Decreasing the temperature of perpendicular rolling down to –30°C leads to a deformation induced martensitic transformation.

Rolling in one pass induces the  $\gamma \rightarrow \alpha'$  transformation and the martensite plates formed within an individual grain exhibit a tendency for gaining a preferred orientation along a common line. The habit plane of the plates does not correspond to the shear plane and remains in a different position in various grains. Perpendicular rolling below the temperature of the beginning of deformation induced martensitic transformation  $M_D$  leads to the formation of transcrystalline martensite plates. The volume fraction of martensite

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formed depends on strain and temperature of deformation.

Martensite developed only by cooling in liquid nitrogen exhibits different morphology from that formed by deformation and is similar to that of quenched steel. The Kurdiumow-Sachs relation between austenite and deformation induced martensite is fulfilled [9]. The annealing at 550 °C of perpendicularly rolled Fe-30Ni alloy leads to the reversed martensite transformation, while subsequent cooling in liquid nitrogen results in the heredity of the structure and the mechanical properties of the alloy achieved by deformation [10]. In the present work the influence of deformation path applied, strain as well as deformation temperature on the morphology and the volume fraction of martensite formed in Fe-30Ni alloy was investigated.

It was found that the influence of the temperature of deformation on the progress of  $\gamma \rightarrow \alpha'$  transformation is more pronounced than the influence of strain value.

Rolling at the temperature range  $M_D - M_S$  (where  $M_S$  is the temperature of beginning of martensitic transformation induced by cooling) increases the driving force of the martensitic transformation, when the temperature approaches  $M_S$ . The volume fraction of martensite increases proportionally with decreasing of the temperature, what influences the properties of the alloy investigated.

## 2. Material and experimental methods

Chemical composition of the alloy investigated is given in Table 1 (in wt.%). The alloy was produced by vacuum melting. After hot working, 8 mm thickness sheets were heat treated by solution annealing at temperature 1150 °C for 1 hour, followed by air cooling to RT. Such solution treated specimens were used for the subsequent experiments.

The martensitic transformation of metastable austenite in Fe-30Ni alloy was obtained by:

- low temperature deformation according different variants of plastic deformation,
- cooling down to liquid nitrogen temperature after plastic deformation.

Plastic deformation was applied by monotonic rolling (realized in one path) as well as perpendicular rolling, with the primary path realized in RT, and the secondary path rotated by 90° around primary longitudinal rolling direction, realized below  $M_D$ .

The aim of this deformation was to achieve a composite-like microstructure. The following variants of thermo-mechanical treatment were performed:

- A. cooling in liquid nitrogen without any plastic deformation,
- B. rolling with 30% strain at –30 °C,
- C. rolling with 30% strain at 20 °C + perpendicular

Table 1. Chemical composition of the Fe-30Ni alloy investigated (wt.%)

C	Mn	P	S	Cu	Cr	Ni	Fe
0.01	0.11	0.007	0.013	0.04	0.38	28.5	bal.

rolling with 20% strain at –30 °C,

D. rolling with 30% strain at 20 °C + perpendicular rolling with 30% strain at –30 °C,

E. rolling with 30% strain at 20 °C + perpendicular rolling with 30% strain at –60 °C,

F. rolling with 30% strain at 20 °C + perpendicular rolling with 30% strain at –80 °C.

The specimens deformed according variants D, E and F were subsequently cooled in liquid nitrogen to complete the martensitic transformation. A quantitative stereological description was performed for these variants to estimate the volume fractions of retained austenite and partial volume fractions of martensite  $V_V^{MF}$ , formed during deformation along different deformation directions. The following partial volume fractions of martensite, connected with different deformation directions, were distinguished:

- main fraction, connected with main deformation direction  $V_V^{MG}$ ,
- ancillary fraction  $V_V^{MP}$ ,
- disoriented fraction  $V_V^{MZ}$ , covering all the remaining directions.

The results of the quantitative microstructural investigations were correlated with mechanical properties measured in tensile tests, namely the tensile strength  $R_m$ , the yield strength  $R_e$ , the total elongation  $A_5$  and the uniform elongation  $A_r$ .

## 3. Results and discussion

The Fe-30Ni alloy in the as-received condition exhibits single-phase austenitic microstructure. To investigate the stability of austenite, the alloy was rapidly cooled down to the temperature of liquid nitrogen. After such cooling a martensitic transformation took place. The high degree of the transformation was confirmed by the relatively low volume fraction of retained austenite, which was determined as 18%. The microstructure of the cooling induced martensite (variant A) was similar to that one of quenched steel, as shown on Fig. 1.

The parameters of the thermo-mechanical treatments were selected on the basis of characteristic temperatures of phase transformations, determined by magnetic methods [11]. The start and finish temperatures of martensitic transformation induced by rapid cooling were determined as  $M_S = -90$  °C and  $M_F = -170$  °C, respectively. The start and finish tem-

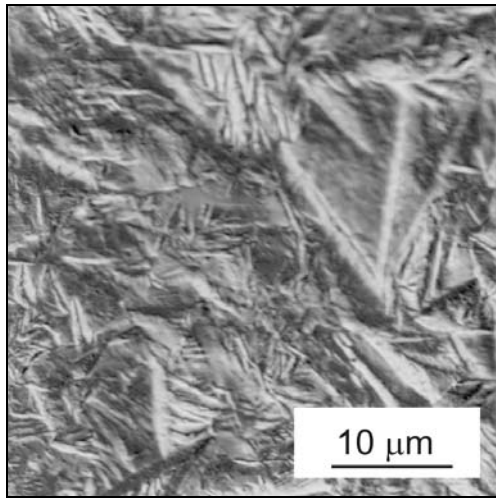


Fig. 1. Microstructure of cooling induced martensite after thermo-mechanical treatment according to variant A. LM.

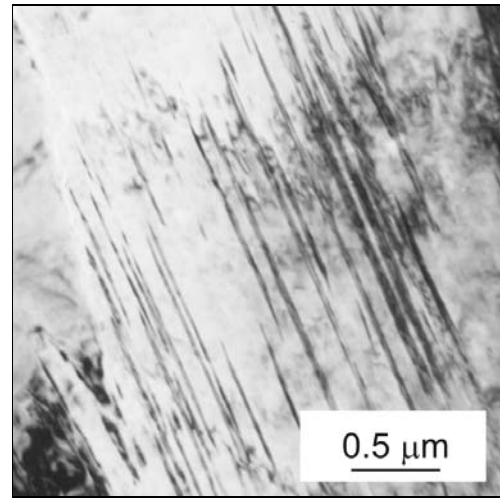


Fig. 3. TEM micrograph of deformation-induced martensite after deformation according to variant B. Martensite plates are in a twin relation.

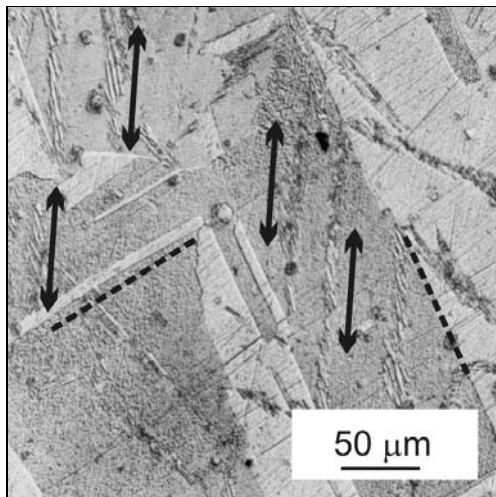


Fig. 2. Microstructure of Fe-30Ni alloy after thermo-mechanical treatment according to variant B. Preferred orientation of martensite plates along the common line ( $\leftrightarrow$ ) and habit planes (---) are marked. LM.

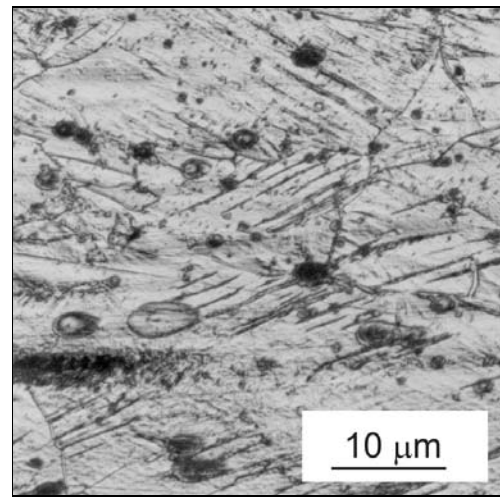


Fig. 4. Transcrystalline martensite needles in Fe-30Ni alloy deformed according to variant C.

peratures of reversed martensitic transformation were measured as  $A_S = 350^\circ\text{C}$  and  $A_F = 450^\circ\text{C}$ , respectively.

Systematic rolling experiments and subsequent magnetic analysis enabled the determination of the  $M_D$  temperature. To induce martensitic transformation by plastic deformation, the starting temperature of rolling equal to  $M_D = -30^\circ\text{C}$  was selected.

Rolling in one path with 30% strain at a temperature  $-30^\circ\text{C}$ , according to variant B, induces the martensitic transformation and the martensite formed exhibits a pronounced preferred orientation of plates along a common line. The habit plane lies on different directions in various grains (Fig. 2). The microstruc-

tural analysis by TEM revealed small single plates inside martensite needles. The needles have linear arrangement dependent on the particular orientation of each austenite grain. However, the single plates inside needles are related to the deformation and lie along the direction of the shear bands formed during rolling at room temperature. Diffraction analysis showed a twin relation between plates (Fig. 3) [12]. The austenite volume fraction was 5.1% for the variant B.

The best tensile properties of the composite-like Fe-30Ni alloy are attained when the deformation induced transcrystalline martensite plates formed are associated with one system of slip bands in austenite. It can be achieved by thermo-mechanical treat-

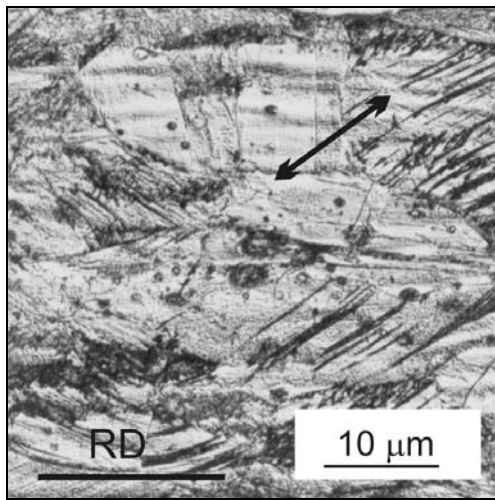


Fig. 5. Morphology of martensite after deformation according to variant D. Main deformation direction ( $\leftrightarrow$ ) and rolling direction (RD) are marked.

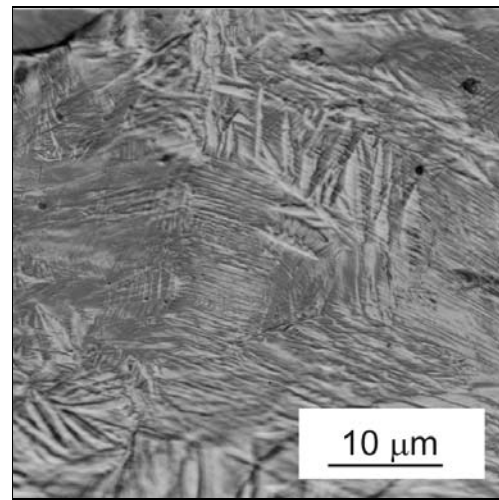


Fig. 7. Microstructure after deformation according to variant D followed by cooling in liquid nitrogen.

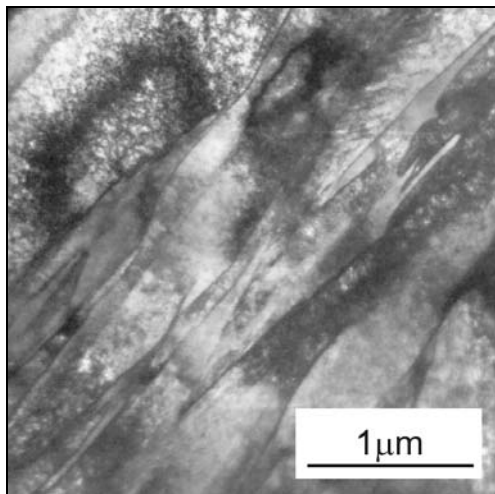


Fig. 6. TEM micrograph of the Fe-30Ni alloy microstructure after deformation according to variant D.

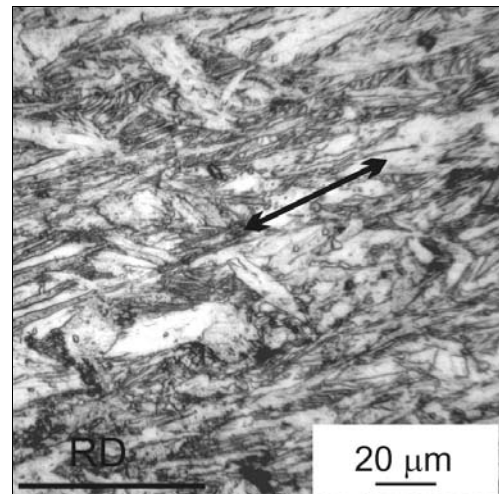


Fig. 8. Transcrystalline martensite plates aligned along the common direction ( $\leftrightarrow$ ) after rolling according to variant E. Rolling direction (RD) is also marked.

ment in the temperature range  $M_D-M_S$ . Perpendicular rolling according to the variant C leads to the formation of transcrystalline martensite needles (Fig. 4). The martensite volume fraction determined after this treatment is 5.3%.

The increase of strain up to 30%, without changing the remaining parameters of thermo-mechanical treatment in variant D, increases the martensite volume fraction up to 7.3%. More than 90% of total martensite fraction is transcrystalline and aligned along the main deformation direction (Fig. 5). TEM micrograph of the Fe-30Ni alloy microstructure after treatment D is shown in Fig. 6. Cooling in liquid nitrogen according to the variant D leads to the completing of the

martensitic transformation, up to 87% of martensite volume fraction. The martensite exhibits a morphology similar to that one of quenched steel (Fig. 7).

It was observed, that the influence of temperature on the martensite volume fraction is much stronger than the influence of the magnitude of applied strain. Therefore, the perpendicular rolling was performed subsequently at temperatures  $-60^\circ\text{C}$  and  $-80^\circ\text{C}$  without changing of other parameters of the thermo-mechanical treatment.

After deformation according to the variant E, with perpendicular rolling at  $-60^\circ\text{C}$ , the martensite volume fraction was 50%. The microstructure was characterized by transcrystalline martensite plates aligned

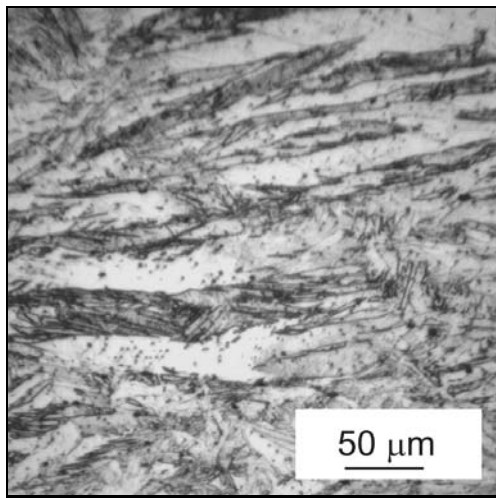


Fig. 9. Microstructure of Fe-30Ni alloy after deformation according to variant F.

along the common direction. High stresses produced by deformation influence the formation of martensite in other directions, as shown in Fig. 8. Cooling in liquid nitrogen after the deformation scheme E leads to the completing of the martensitic transformation, up to 90% of martensite volume fraction.

Decreasing the temperature of perpendicular rolling down to  $-80^{\circ}\text{C}$  in variant F results in increasing of martensite volume fraction up to 80% (Fig. 9). The tiling of plates along one direction still dominates, however the volume fractions of martensite formed in other directions increase [13]. Figure 10 shows the TEM micrographs of the microstructure after rolling according to the variant F. Subsequent cooling in liquid nitrogen increases the martensite volume fraction after deformation in variant F up to 92%.

The results of the mechanical properties of Fe-30Ni alloy after thermo-mechanical treatment are given in Table 2. After cooling in liquid nitrogen without deformation, the mechanical properties are worse than after deformation. The increase of the martensite volume fraction associated with the main deformation direction, responsible for composite-like character of the alloy, results in increasing the strength of Fe-30Ni alloy.

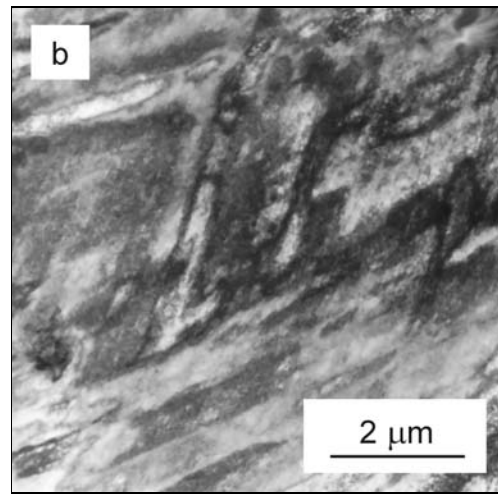
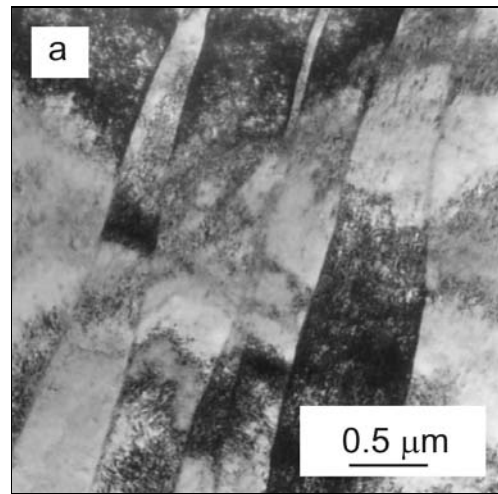


Fig. 10a,b. TEM micrographs (scale: (a)  $0.5\ \mu\text{m}$ , (b)  $2\ \mu\text{m}$ ) of the microstructure after rolling according to variant F.

#### 4. Conclusions

1. A composite-like microstructure has been produced in the Fe-30Ni alloy.
2. It was found that the influence of the temperature of deformation on the progress of  $\gamma \rightarrow \alpha'$  transformation is more pronounced than the influence of strain magnitude itself.
3. Rolling in the temperature range  $M_D - M_S$  increases the driving force inducing the martensitic

Table 2. Mechanical properties of Fe-30Ni alloy after selected variants of thermo-mechanical treatment

Variant	$R_m$ (MPa)	$R_e$ (MPa)	$A_5$ (%)	$A_r$ (%)	Martensite $V_V$ (%)
A	814.8	728.9	7.1	4.7	82
D	895.0	699	5.1	3.0	88
E	914.1	729	5.2	2.5	90
F	916.2	731	5	2.3	92

transformation, when the temperature approaches  $M_S$ . The volume fraction of martensite increases proportionally with decrease of the temperature, what influences the properties of the alloy investigated.

4. The partial volume fraction of martensite formed along the main deformation direction is mainly responsible for hardening of the alloy.

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